CHEMICALLY ACTIVATED CARBON USING PEAT SOIL FOR HEAVY METALS ADSORPTION IN INDUSTRIAL WASTEWATER

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A Thesis submitted in fulfilment of the
requirement for the award of the degree of
Masters of Science

Faculty of Applied Sciences and Technology
Universiti Tun Hussein Onn Malaysia

JANUARY 2019

For my beloved mother, Dalina binti Kariban. My late father which I miss every day, Allahyarham Md Jamien bin Salman, family members and friends. Not forgetting my best friend Khawarizmi bin Mohd Jafery and Abdul Jalil bin Abdul Rahim who have continuously supporting me all time long.

ACKNOWLEDGEMENT

The author would like to express his sincere appreciation to his inspiring and desirable Project Supervisor, Assoc. Prof. Dr. Zaidi Bin Embong and his wife, Pn Salawati Binti Ahmad for the support given throughout the duration for this research.

The cooperation given by the Faculty of Mechanical and Manufacturing Engineering (FKMP), Faculty of Civil and Environment Engineering (FKAAS) and Microelectronics and Nanotechnology – Shamsuddin Research Center (MiNT – SRC), Universiti Tun Hussein Onn Malaysia (UTHM) are also appreciated for providing the consultancies, equipments and space to run out the analysis. Appreciation also goes to everyone who has been involved directly and indirectly towards the compilation of this thesis. A thousand thanks to Research Centre for Soft Soil (RECESS), UTHM for providing a working station to do this research.

ABSTRACT

The present work is aimed to produce peat soil activated carbon (PSAC) for the removal of heavy metal ions in industrials wastewater. Activated carbon from peat has successfully produced by chemical activation process using sulphuric acid as activating agents. The effect of heating temperature on the carbon content, the physical and chemical characterizations of peat activated carbons were studied. From this study, peat soil heated at 400℃ possess the highest carbon content with concentration of 95.44 % due to the complete degradation of organic matter. The thermogravimetric analysis (TGA) and its derivative thermogravimetric (DTG) confirm the finding where at temperature ≈400°C, peat undergo complete degradation of matter. The heating temperature was set to constant at 400°C for activation process. The BET specific surface area of the activated carbon obtained was 1298.84 $m^2 g^{-1}$. The Raman spectrum gave to main peaks at 1368 and 1590 cm^{-1} which is attributed to diamond, D and graphitic, G band, respectively. FTIR spectrum analysis showed the carboxyl, ketone, aldehyde, methylene and hydroxyl groups are present on the surface of PSAC due to the activation process with H_2SO_4. Point of zero charge (pH_{pzc}) of activated carbon is 5.60 which available for heavy metal adsorption due to the adsorbent surface charge was negative. The heavy metal adsorption capacity is evaluate by using an adsorption test. The heavy metal elemental compositions (Cu, Zn, Al, Cr, Pb and Fe) in the industrial wastewater were analysed using ICP – MS. The adsorption test was conducted using batch experiment method where various dosage of PSAC (0.0, 0.2, 0.4, 0.6, 0.8, 1.0 g) mixed to 25 ml water samples. From the adsorption test, the PSAC can be used as adsorbent for industrial waste water treatment.

ABSTRAK

Kajian terikini bertujuan untuk menghasilkan karbon teraktif tanah gambut bagi penyingkiran ion-iot logam berat yang terkandung dalam air sisa industri. Karbon teraktif yang terhasil dari tanah gambut ini berjaya dihasilkan melalui kaedah rawatan pengaktifan kimia yang menggunakan asid sulfurik sebagai bahan pengaktifan. Antara kajian yang dijalankan dalam penyelidikan ini adalah kesan suhu pemanasan terhadap kandungan karbon dalam tanah gambut, dan percirian fizikal dan kimia karbon teraktif. Dalam kajian ini, tanah gambut yang dipanaskan pada suhu 400°C menghasilkan kandungan karbon yang amat tinggi iaitu sebanyak 95.44%, yang disebabkan oleh penguraian lengkap bahan organik. Analisis termogravimetri (TGA) dan derivatifnya (DTG) mengesahkan dapatan tersebut dimana penguraian lengkap berlaku pada suhu sekitar 400°C. Suhu pemanasan tersebut adalah ditetapkan sebagai pemalar bagi proses pengaktifan. Luas permukaan spesifik BET karbon teraktif adalah 1298.84 m^2 g^{-1}. Spektrum Raman menunjukkan puncak utama pada 1368 dan 1590 cm^{-1} yang masing-masing mewakili jalur berlian, D dan grafit, G. Analisis spectrum inframerah FTIR menunjukkan bahawa kumpulan karboksil, ketone, aldehida, metilena dan hidroksil terhasil pada permukaan yang terjadi semasa proses pengaktifan Bersama H_2SO_4. Nilai titik bercaj sifar (pH_{pzc}) karbon teraktif adalah 5.60 dimana sesuai untuk serapan logam berat dimana caj permukaan bahan serapan adalah negatif. Ujikaji penyerapan dijalankan untuk mengkaji muatan serapan logam berat. Logam berat (Cu, Zn, Al, Cr, Pb and Fe) dalam spesimen air sisa industri dianalisis menggunakan ICP – MS. Ujikaji penyerapan ini dijalankan dengan dos karbon teraktif iaitu jisim bahan bagi setiap 25 ml spesimen air. Maka hasil dari kajian ini, karbon teraktif terhasil dari tanah gambut sesuai digunakan sebagai bahan penyerap untuk merawat air sisa industri.

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LIST OF SYMBOLS AND ABBREVIATIONS

AC	-	Activated carbon
Al	-	Aluminium
As	-	Arsenic
at %	-	Atomic percentage
C	-	Carbon
C_e	-	Equilibrium concentration of adsorbate in solution
CO	-	Carbon monoxide
CO_2	-	Carbon dioxide
COOH	-	carboxyl
COT	-	Carbon extracted at an optimum temperature
Cr	-	Chromium
Cu	-	Copper
D - band	-	Diamond band
Fe	-	Iron
FTIR	-	Fourier Transformed Infrared Spectroscopy
G - band	-	Graphitic band
H_2SO_4	-	Sulphuric acid
H_3PO_4	-	Phosphoric acid
HCl	-	Hydrochloric acid
HNO_3	-	Nitric acid
ICP - MS	-	Inductively Coupled Plasma – Mass Spectrometry
I_D	-	Intensity of diamond band
I_G	-	Intensity of graphitic band
K_2CO_3	-	Potassium carbonate
K_2O	-	Potassium oxide

KBr	-	Potassium bromide
keV	-	Kilo electron volt
m	-	Mass of specimen
$m^2\ g^{-1}$	-	Squared meter per gram
$m^3\ h^{-1}$	-	Cubic meter per hour
$mg\ g^{-1}$	-	Milligram per gram
$mg\ L^{-1}$	-	Milligram per litre
N_2	-	Nitrogen gas
N_A	-	Avogadro’s number
NaOH	-	Sodium hydroxide
Ni	-	Nickel
nm	-	Nanometer
O_2	-	Oxygen
OH	-	hydroxyl
P	-	Phosphorus
P	-	Equilibrium pressure
P_0	-	Saturation pressure
Pb	-	Lead
pH_{pzc}	-	Point of zero charge
pH_{sol}	-	pH of solution
ppb	-	Part per billion
PSAC	-	Peat soil activated carbon
PSAC - PA	-	Peat soil activated carbon after adsorption test (post adsorption)
q_e	-	Metal uptake capacity
q_m	-	Maximum capacity of adsorbent
R	-	Intensity ratio
s	-	Molecular cross section of adsorbing materials
S_{BET}	-	Specific surface area
SEM - EDX	-	Scanning Electron Microscope – Energy Dispersive X – Ray spectroscopy
Si	-	Silicon
S_{total}	-	Total surface area
v	-	Adsorbed gas quantity

v_m	-	Monolayer adsorbed gas quantity
Zn	-	Zinc
$ZnCl_2$	-	Zinc chloride
°C min^{-1}	-	Degree Celcius per minute
μm	-	micrometer

CHAPTER 1

INTRODUCTION

1.1 Background of study

Economic bloom and high living standards causes the industrial revolution to increase. Rapid industrialization causes the heavy metal contaminants being discharged into the environment from their wastewater (Amuda, Giwa, & Bello, 2007a). One of the key topics that have attracted attention globally is the water contaminant released from industrial wastewater due to the negative impact in environment and biological systems (Sabela et al., 2016). Heavy metal pollution may come from many industries such as electronic fabrications, paints and pigments and manufacturing processes. Wastewater commonly includes aluminium, chromium, lead, zinc, iron and copper (Amuda, Giwa, & Bello, 2007b;Salam, Reiad, & Elshafei, 2011).

Al compound is shown to be neurotoxic if it enters the bloodstream. Bone mineralization disorder and defects of the brain may be occur for the people who receive an excessive exposure of Al (Al-Muhtaseb, El-Naas, & Abdallah, 2008). Cr is suspected to be a carcinogenic as it affects the kidney, liver and causes diarrhea and dermatitis (Rai et al., 2016). According to El-Wakil, El-Maaty, & Awad, (2014), Pb is the most toxic element even in a low concentration. Pb toxicity may lead to the nephritic syndrome, hepatitis, anaemia and several diseases such as nervous system, kidney and liver problems. Zn is essential to physiological functions of living tissue and regulate many bio-chemical processes. However, excessive consumption of Zn might cause vomiting, kidney and stomach damage, and skin problems (Amuda et. al., 2007b). Fe is essential for human as it plays a main role for oxygen transportation in red blood cell. But there are some consideration that makes water is un-usable like metallic taste, stains and odour (Myalowenkosi, Kwanele, & Suvardhan, 2015). In contrast, the Fe may precipitate a dark sludge when Fe in water exposed to air in atmospheric pressure and becomes oxidised or ferric form that will not dissolved in water, and influence the ferruginous bacteria resulting in neurological disorder (A. Akl, Yousef, & AbdElnasser, 2013). Heavy metal such as Cu tends to accumulate in living organism resulting various diseases and health disorder. The excessive exposure of Cu whether direct or indirect causes Cu to accumulate in the liver and produces gastrointestinal problems (Imamoglu & Tekir, 2008). Because of the above factors, there is a significance to remove these metals from wastewater in conjunction with prevention of metal pollutant by effluents of toxic properties.

There are several ways to remove heavy metal composition from wastewater such as oxidation (Gunatilake SK, 2015), reverse osmosis (Aljendeel, 2011), membrane filtration (Qdais & Moussa, 2004) and adsorption (I. A. W. Tan, Chan, Hameed, & Lim, 2016).

Adsorption of heavy metal on activated carbon conforms to be the effective and efficient technique in this application (Myalowenkosi et al., 2015). Adsorption of heavy metal on adsorbents such as activated carbon has been used as an effective adsorbent. Activated carbon has unique characteristics such as highly developed porosity, large internal surface area, surface charge, surface functional groups and the adsorption capabilities of metal ion in activated carbon (El-Wakil et al., 2014).

There are several ways to produce activated carbon such as physical, chemical and also microwave assisted activation method. Physical activation method refers to a mechanism that include pyrolysis process under atmospheric pressure, followed by an activation process using oxidizing gas such as steam, CO_2, N_2 or air at high temperature ranges from 800 to 1100°C. On the other hand, chemical activation process is a process that includes mixing step of chemical agent and precursor upon heat treatment in at temperature ranges from 400 to 600°C under atmospheric condition. High carbon yield obtained from chemical activation method compared to physical one (Rashidi & Yusup, 2017). It provides economical viable as it has lower activation temperature and shorter processing time. Additionally, activated carbon prepared from microwave heating also undergoes chemical treatment. Microwave heating approach was used as it provide rapid heating rate compared to conventional electric furnace. However, there were different in physiochemical properties of activated carbon from microwave heating where the surface area of activated carbon using furnace was exceptionally higher than microwave assisted (Rashidi & Yusup, 2017).

Generally, raw materials from various agricultural by–products have been used such as tamarind seed (Mopoung, Moonsri, Palas, & Khumpai, 2015), oil palm bunch (Wahyuningsih & Yusri, 2016), olive stone (Gautam et al., 2015), rice husk (Manoj Kumar Reddy, Krushnamurty, K Mahammadunnisa, Dayamani, & Ch Subrahmanyam, 2014), coconut button (Anirudhan & Sreekumari, 2011), apple wastes (Hesas, Arami-Niya, Mohd, Daud, & Sahu, 2013) and jackfruit peel (Prahas, Kartika, Indraswati, & Ismadji, 2008).

From those raw materials, peat is one of the rich sources of carbon and abundant. Compared to other soils such as acrisols and fluvisols, peat has the greater carbon content. Acrisols is a kaolinitic-content soil which has mean pH of 4.5 and high mineral salt element such as Mg, Ca, Na and K (Tho & Hoa, 2017). On the other hand, fluvisols is an acid-sulphate soils which dominant in sea sediment. Fluvisols has greater amount of Al with low pH value of 3.5 (Kluiving, De Ridder, Van Dasselaar, Roozen, & Prins, 2016). Acrisols and fluvisols have low carbon content. Thus peat offer little economic value and wide range of applications. The carbon content from peat is contributed from the decaying plant residues. Furthermore, peat has high carbon and organic matter that make it important for metal uptake by the sorbent. Thus, peat can be modified in order to improve the sorption capacity since biosorption of peat is inexpensive and large spectrum of applications. Peat also consists of variety of surface functional groups such as hydroxyl, lactone, phenolic and carboxyl groups that plays a main role in complexation and metal ion exchange during ion fixation (Batista et al., 2009; Hartman, Chen, & Hatcher, 2015; Xiong & Mahmood, 2010). The study of activated peat as adsorbent material for heavy metal adsorption is still lack and less information could be obtained on the adsorption properties. Thus, the study on activated peat from different activation method, type of pollutant and activating agent. The previous work on peat will be discussed in Literature Review.

1.2 Problems statement

The industrial wastewater such as electronic factories contribute the heavy metal contaminants that may lead to hazardous effect for biological life and environment. Wastewater polluted by heavy metal should undergo treatment before being discharged into the drainage systems. Adsorption method using activated carbon is one of the effective way to remove heavy metal contaminants in wastewater (Kumar

& Jena, 2017). However, the materials used as precursors have different capability to adsorb different type of metal ions due to its high adsorption capacities and the functional groups on the surface of activated carbon (Sabela et al., 2016). The effective and inexpensive technologies capable of treating wastewater rich in metal contaminants and biosorption has emerged as a viable technique for metal removal. Biosorption offers advantages including minimal production of chemical or biological sludge and efficient removal and no nutrient requirement (Batista et al., 2009). In previous work on peat as adsorbent for metal removal in wastewater, there are some activation reagent used to activate peat such as HCl (Batista et al., 2009), H_2PO_3 (Mudjijati et al., 2000), iron coated peat (Kasiuliene et al., 2018) and $ZnCl_2$ (Varila et al., 2017). However in this study, H_2SO_4 was used as an activating agent. In previous study on metal sorption by activated peat, less type of metal concerned such as Cr, Cu, Ni, Cd, and As. However, in present work, a several type of metals that has different kind of toxicity such as Al, Fe, Zn and Pb will be study and comprehend their sorption capability.

1.3 Objectives

The aim of this research is to generate activated carbon from peat soil activated with sulphuric acid for adsorption of heavy metal contaminants. To achieve the aim of the research, a study was carried out with the following objectives:

1. To determine the optimum heating temperature of carbon extraction from peat soil.
2. To determine the graphitic contents and functional group on peat soil activated carbon.
3. To evaluate the adsorption efficiency of heavy metal contaminants from wastewater on peat soil activated carbon.

1.4 Significance of study

Activated carbon is used as an adsorbent to remove organic and inorganic pollutant from liquid and gas phase. Activated carbon is used in the wastewater treatment of variety heavy metal contaminants. The development of activated carbon in industrial scales could approach towards controlling the discharge of heavy metal contaminants hence reducing water pollution. The adsorption of heavy metal contaminants using activated carbon were done from previous research with significant outcome. However, the study on chemically prepared peat soil activated carbon for metal adsorption is lack. A study reported by Batista et al., (2009) found that the activation agent used was HCl and this study was focus on Cr adsorption. There were no varieties of the chemical agents and metal species. Another study on peat soil activated carbon reported by Mudjijati et al., (2000), shows the production of activated carbon was success but there were no study on metal species except more focus on its characterisation. At a glance, the studies on the metal adsorption using activated carbon were using standardised initial concentration of metal contaminant which was synthetically produced in laboratories.

This study has its betterment of research area in terms of the initial concentration of metal contaminant. The contaminated wastewater was taken from the selected industrial parks which provide actual concentration and varieties metal elements from the real polluted environment. The heating temperature of peat soil were manipulated as well as to determine the optimum temperature in order produce high carbon concentration. Next, the comparison of graphitic content in raw peat soil, peat soil heated at optimum temperature and peat soil activated carbon were conducted in this study.

1.5 Scopes of the study

This research was conducted in order to produce activated carbon prepared from peat for the removal of metal contaminants in industrial wastewater. The aspects looked into were the number of sampling points (5 sampling point at Kg Pt Nipah, Senggarang), heavy metal contaminants in wastewater, facilities used for investigation and analysis, and method used during this study. Some limitations for this study are as follow;

1. Heating up peat soil as precursor from 200℃ to 700℃. Chemical activation method was used to produce peat soil activated carbon.
2 H_2SO_4 was used as activating agent with mixing ratio 1:1 and heated up to 400°C for an hour at atmospheric pressure.
3. This work also focuses on the physical and chemical characteristics of activated peat.
4. Chemical characteristics of activated peat would be analysed using Laser Raman spectroscopy for graphitic content and FTIR spectroscopy for the surface functional groups identification.
5. Industrial waste water specimen would be taken from three different industrial parks which were located at Sri Sulong, Sri Gading and Parit Raja industrial park. The water specimen was taken using physical method as describe in ISO 5667. The heavy metal in this study would be focused on Al, Cr, Pb, Zn, Fe and Cu. These metals are analysed using ICP-MS.
6. This study also carried out the adsorption test using different dosage (0.0, 0.2, 0.4, 0.6, 0.8, 1.0 g) of activated peat for every 25 ml of waste water. The removal efficiency analysis conducted using the remaining (final) concentration of heavy metal in water specimens.
7. Langmuir and Freundlich adsorption isotherm would be used for adsorption capacity on different type of metal species.

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